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Summary of the 1983 EPA (Environmental Protection Agency) National Performance Audit Program on Source Measurements

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16. ABSTRACT

In the spring and fall of 1983, the Quality Assurance Division conducted the National Audits for Stationary Source Test Methods. The audit materials consisted of a calibrated orifice for Method 5 (dry gas meter only), five simulated liquid samples each for Method 6 (SO_2) and Method 7 (NO_x), two coal samples for Method 19A, and a disposable gas cylinder for Method 3 (Orsat analyzer). Participating laboratories sent their data to the Source Branch and in return received a written report comparing their results to EPA's.

In the Method 5 spring audit, the mean value for all participants differed by 5.7% from the true (EPA) value. For the fall audit, the participants' mean was 4.1% from the true value. In the two Method 6 audits, the average mean differed by 3% from the expected value. The average mean in the two Method 7 audits was 15% from the expected value.

In the two coal audits, the parameters measured were sulfur, moisture, ash, and Btu. On the average for the sulfur analysis 85% of the participants measured within 10% of the expected value; for Btu 100% of the participants measured within 10% of the expected value.

In the two Method 3 audits, each parameter had only one concentration. The means for CO_2 were within 10% (spring) and 6% (fall) from the expected value. For both audits, the mean for O_2 was less than 1%.

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the environment, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection regulations through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems; implementation of agency-wide quality assurance programs for air pollution measurement systems; and supplying technical support to other groups in the Environmental Protection Agency, including the Office of Air and Radiation, and the Office of Toxic Substances.

The major task of this study was to report the results of the national quality assurance audit program for stationary source test methods. Audits were designed to estimate the minimal analytical and computational accuracy that can be expected with EPA Method 5 (dry gas meter only), Method 6 (sulfur dioxide), Method 7 (nitrogen oxides), Method 19 (coal), and Method 3 (carbon dioxide and oxygen). Statistical analysis was used to characterize the data.

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ABSTRACT

In the spring and fall of 1983, the Quality Assurance Division conducted the National Audits for Stationary Source Test Methods. The audit materials consisted of: a calibrated orifice for Method 5 (dry gas meter only), five simulated liquid samples each for Method 6 (SO_2) and Method 7 (NO_x), two coal samples for Method 19A, and a disposable gas cylinder for Method 3 (Orsat analyzer). Participating laboratories sent their data to the Source Branch and in return received a written report comparing their results to EPA's.

In the Method 5 spring audit, the mean value for all participants differed by 5.7% from the true (EPA) value. For the fall audit, the participants' mean was 4.1% from the true value. In the two Method 6 audits, the average mean differed by 3% from the true value. The average mean in the two Method 7 audits was 15% of the true value.

In the two coal audits, the parameters measured were sulfur, moisture, ash, and Btu. On the average for the sulfur analysis, 85% of the participants measured within 10% of the expected value; for Btu, 100% of the participants measured within 10% of the expected value.

In each of the two Method 3 audits, each parameter had only one concentration. The means for CO_2 were within 10% (spring) and 6% (fall) of the expected value. For both audits, the mean for O_2 was less than 1%.

This report covers the period from January 1983 to December 1983.

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We express our appreciation to the laboratories that participated in our audits. Thanks also to the Standards Laboratory (EPA/EMSL/ Performance Evaluation Branch) who did our Acceptance Testing on the audit samples. For providing the data systems to store and evaluate the data, we thank the programmers of the Data Management and Analysis Division/EMSL.

SECTION 1

INTRODUCTION

The Environmental Monitoring Systems Laboratory (EMSL) of EPA, in 1977, established a performance audit program to evaluate the performance of companies that conduct compliance testing, using EPA Reference Methods. The audits verify the analytical accuracy of EPA Reference Methods 3, 6, 7, and 19A and the calibration accuracy of the Method 5 control console (1). Accuracy is defined as the percent difference between the participant's analytical results and the EPA expected value. By participating in this (free and voluntary) program, testing companies can compare their performance to other laboratories conducting similar measurements.

Two audits were conducted for each of Methods 3, 5, 6, 7 and 19A in 1983. Every participating laboratory received an audit package consisting of the audit sample, a data card, instructions, and an envelope for returning the data to EPA. A label for returning the audit device was also included for the Method 5 audits. Participants had 8 weeks to return data to EPA. At the end of this period all data received were statistically analyzed to determine the precision and accuracy obtained by the participants. Participants were given 8 weeks to return the data, which was then statistically analyzed for precision (repeatability) and accuracy.

The Quality Assurance Division of EMSL maintains a repository of audit samples for the EPA Methods 3, 6, 7 and for coal. These (stable) samples are available to any laboratory needing them (for example) to train new personnel or to conduct quality control checks in the laboratory. Since the expected values (for these samples) are included with the analysis instructions, there is no requirement for the data to be returned to the EPA. However, we recommend that participants make use of this sample repository to help improve their overall analytical skills.

This report summarizes the results obtained in the 1983 source audits.

SECTION 2

SUMMARY

In the spring and fall of 1983, EPA's EMSL, Research Triangle Park, North Carolina, conducted National Quality Assurance Audits for Stationary Source Test Methods 5 (dry gas meter only), 6 (SO_2), 7 (NO_x), 19A (coal), and 3 (Orsat analyzer). Industrial laboratories, contractors, foreign laboratories, as well as local, state, and federal agencies participated.

Two Method 5 audits were conducted in 1983. The overall results (no outliers removed) are summarized in Table 1. In the first audit, the mean for all participants was 5.7% from the true value and in the second audit it was 4.1%. After correcting for outliers, the means for the 0383 and 0983 audits were 2.6% and 2.1%, respectively, from the true value. The participants' performances were not statistically different from previous audits (2,3,4).

TABLE 1. PARTICIPANTS' RESULTS FOR METHOD 5 AUDITS
(ALL DATA - NO OUTLIERS REMOVED)

Type of sample	Parameter	Audit date	No. of analyses	Mean (% from EPA value)	Median (% from EPA value)	Std. dev.
Orifice	Volume	0383	763	5.7	2.2	32.7
		0983	614	4.1	1.9	21.3

Table 2 presents the data (no outliers removed) from the two 1983 Method 6 audits. This audit procedure requires the participants to determine the sulfate content in five aqueous solutions using the titration procedure of Method 6. For each concentration in the 0283 audit, the mean of the participants was 4% higher than the true value, in contrast, the median differed by less than 1%. In the 0883 audit, the means differed by 3%, and the medians again were less than 1%. In both audits, 50% to 68% of the participants achieved an accuracy within 2% for 9 out of 10 of the samples. However, on one low concentration sample, only 43% achieved this accuracy.

TABLE 2. PARTICIPANTS' RESULTS FOR METHOD 6 AUDITS
(ALL DATA - NO OUTLIERS REMOVED)

Type of sample	Audit date	No. of analyses	EPA true value	Participant results		
				Mean	Median	Std. dev.
Aqueous sulfate	0283	113	343.20	361.54	342.80	109.11
		113	762.60	794.51	758.30	233.13
		113	1143.90	1188.32	1140.00	349.57
		112	1906.50	1971.14	1890.00	580.91
		113	2478.50	2579.04	2456.00	778.94
	0883	88	396.60	393.57	395.50	50.93
		90	594.80	589.35	595.15	67.21
		90	633.00	621.39	629.35	89.04
		90	1349.80	1314.46	1339.45	168.73
		90	1426.10	1384.05	1416.65	192.16

Table 3 presents the data (no outliers removed) from the two Method 7 audits in 1983. This audit procedure requires that the participants determine the nitrate content in five aqueous solutions. For each concentration in the 0483 audit, the mean of the participants was 22% higher than the true value, but in contrast, the median differed by less than 3%. In the 1083 audit, the means were 12% higher than the true value, and again the median value differed by only 3%. In both audits, 34% to 38% of the participants achieved an accuracy within 3% of the true value for 8 out of 10 samples. However, on the two lowest concentration samples only 25% achieved this accuracy.

TABLE 3. PARTICIPANTS' RESULTS FOR METHOD 7 AUDITS
(ALL DATA - NO OUTLIERS REMOVED)

Type of sample	Audit date	No. of analyses	EPA true value	Participant results		
				Mean	Median	Std. dev.
Aqueous sulfate	0483	59	59.70	71.66	58.10	68.67
		62	298.70	348.99	302.80	280.20
		63	477.80	581.76	480.00	579.80
		62	737.20	892.88	750.00	720.34
		63	955.60	1159.22	975.60	1005.48
	1083	53	118.40	128.12	121.00	32.76
		53	218.40	235.98	221.40	64.95
		54	240.30	267.01	245.70	71.78
		54	407.20	440.13	407.45	136.98
		52	454.80	511.48	469.65	143.74

Table 4 summarizes the results of the two coal audits. Participants analyzed each coal sample in duplicate for percentage of sulfur, moisture, and ash, and for gross calorific value (Btu/lb). The means of the ash, moisture, and sulfur content were within 4% of the expected value on both concentrations. An accuracy of 1% was achieved on the Btu content.

TABLE 4. PARTICIPANTS' RESULTS FOR METHOD 19A COAL AUDIT
(ALL DATA - NO OUTLIERS REMOVED)

Type of sample	Audit date	Parameter	No. of analyses	EPA true value	Participants' Results		
					Mean	Median	Std. dev.
Coal	0383	% S	125	0.74	0.74	0.74	0.05
	0983		112	0.74	0.75	0.73	0.21
	0383		127	2.98	2.89	2.91	0.17
	0983		113	2.95	2.85	2.87	0.21
	0383	% H_2O	125	2.05	2.06	2.05	0.71
	0983		113	1.62	1.59	1.68	0.37
	0383		127	6.13	5.97	6.16	0.53
	0983		114	5.75	5.86	6.10	0.71
	0383	% Ash	123	7.40	7.35	7.33	0.41
	0983		112	7.31	7.60	7.29	3.35
	0383		125	11.42	11.37	11.42	0.24
	0983		113	11.33	11.61	11.40	2.59
	0383	Btu/lb	125	12719.00	12643.50	12684.00	251.79
	0983		112	12470.00	12574.22	12597.50	207.38
	0383		123	14165.00	14115.97	14141.00	150.59
	0983		111	14059.00	14068.90	14093.00	170.01

The results of the Method 3 audits are summarized in Table 5. Participants analyzed the gas sample twice for percentage of carbon dioxide, oxygen, and carbon monoxide. The mean value of carbon dioxide in the 0583 audit differed by 11% from the expected value; whereas, in 1183 the mean value was within 6%. In both audits, the mean values for oxygen were within 1%. In contrast, the mean values for carbon monoxide differed as much as 21% from the expected value.

TABLE 5. PARTICIPANTS' RESULTS FOR METHOD 3 AUDIT
(ALL DATA - NO OUTLIERS REMOVED)

Type of sample	Audit date	Parameter	No. of analyses	Replicate	EPA true value	Participants' results		
						Mean	Median	Std. dev.
Small cylinder	0583	% CO_2	64	1	5.00	4.49	4.70	0.73
			63	2	5.00	4.59	4.70	0.98
		% O_2	64	1	15.10	15.12	15.05	1.22
			63	2	15.10	15.10	15.00	1.22
		% CO	53	1	1.00	0.81	0.80	0.30
			53	2	1.00	0.79	0.90	0.33
	1183	% CO_2	52	1	7.10	6.72	6.80	0.34
			51	2	7.10	6.70	6.80	0.53
		% O_2	52	1	11.20	11.23	11.40	1.20
			51	2	11.20	11.20	11.40	1.27
		% CO	40	1	0.50	0.47	0.50	0.23
			40	2	0.50	0.47	0.40	0.31

SECTION 3

METHOD 5 DRY GAS METER AUDIT

In the Method 5 audit procedure, participants use a calibrated orifice to check the calibration of the dry gas meter in their EPA Method 5 control console (meter box). They insert the orifice in the Method 5 meter box, allow the box to warm-up, and then make three 15-minute volume measurements. The participants convert each of the three volumes to cubic meters at standard conditions using the formula specified in Method 5 (Eq. 5.1 of Appendix A 40 CFR 60) and record them on the data card. Finally, they mail the orifice and the data card to EPA for statistical analysis.

In the spring audit (0383), 71% of the 160 laboratories that received the audit package returned data. In the fall audit (0983), 65% of the 153 laboratories returned data. These percentages are slightly lower than those found in previous audits (2,3,4). Table 6 (which classifies the participants into general categories) compares the number of participants who requested to participate in the Method 5 audit with the number who actually returned data.

TABLE 6. METHOD 5 AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0383	0983	0383	0983
Contractor	88	80	59	43
University	1	0	0	0
Industry	44	43	33	32
Foreign	3	4	3	4
Federal	3	3	3	2
State	15	17	12	15
Local	6	6	4	4
Total	160	153	114	100

Figure 1, a cumulative histogram, shows the accuracy obtained by participants in the 0383 and 0983 Method 5 audits, expressed as the percent difference from the true (EPA) value at various levels of accuracy. The Code of Federal Regulations (1) requires that the dry gas meter be calibrated with an accuracy of ± 2%. Figure 1 also shows that 46% of the reporting laboratories in the 0383 audit and 53% in the 0983 audit obtained

this accuracy. These results indicate a slight increase to those in previous audits (Figure 2). Seventy-three of the laboratories participated in both audits.

The histograms in Figures 3 and 4 compare the individual results of the 0383 and 0983 audits with the mean and the median values for all participants. The majority of the laboratories (0983) reported values higher than the EPA value. The standard deviation of the triplicate analysis (precision) by each laboratory indicated that for the 0383 audit, 65% of the standard deviations for each set were within 0.3%. For the 0983 audit, 70% of the standard deviations were within 0.3%. Three percent of the 0383 and 4% of the 0983 data were identified as outliers using Chauvenet's Criterion (5). Before the outliers were removed, the mean values for the 0383 and 0983 data differed by 5.7 and 4.1% from the true value, respectively. After deletion of outliers, these values were reduced to 2.6 and 2.1, respectively.

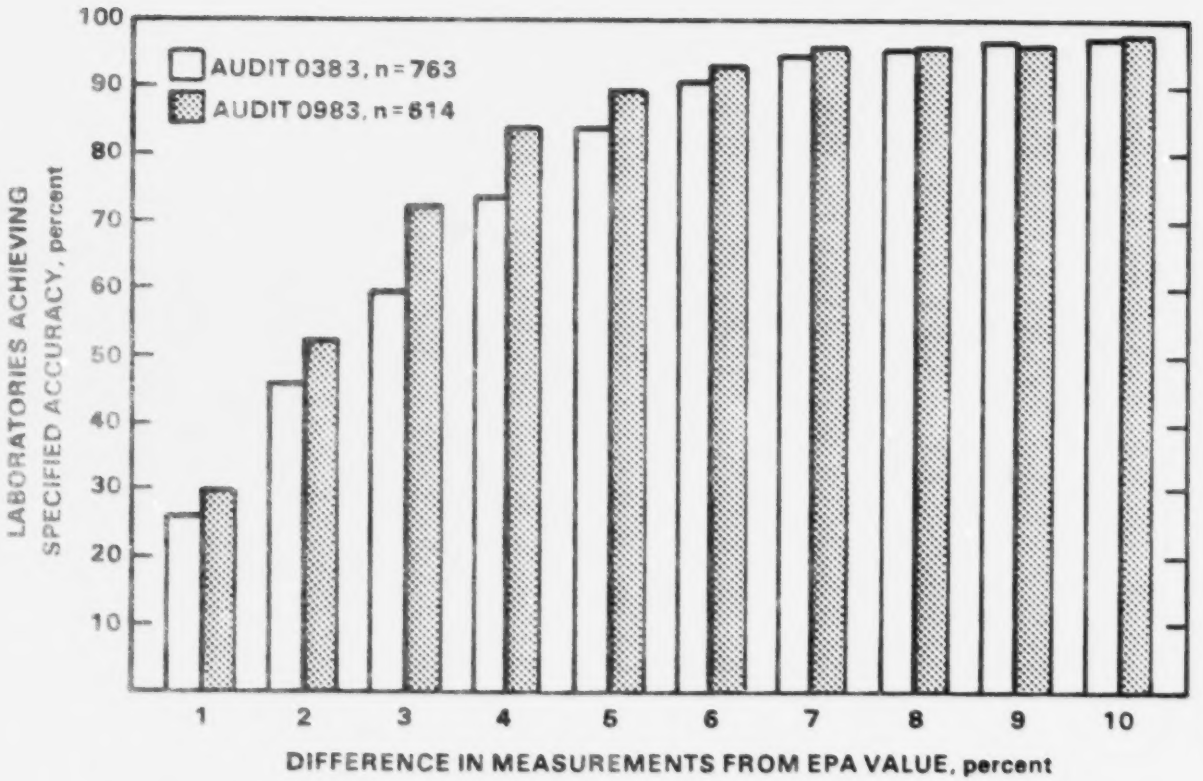


Figure 1. Cumulative accuracy for participants in Method 5 audits 0383 and 0983.

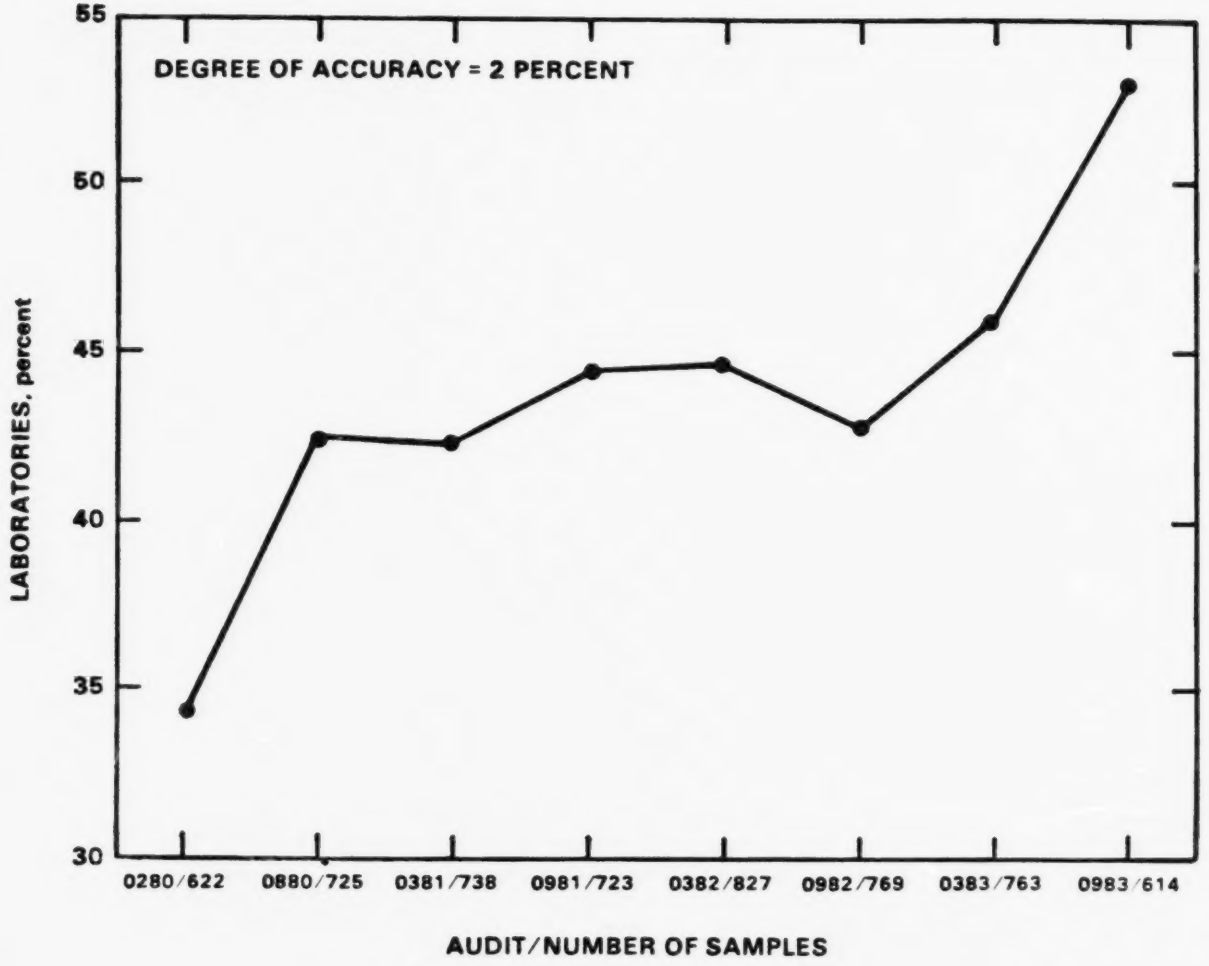


Figure 2. Previous results of Method 5 audit.

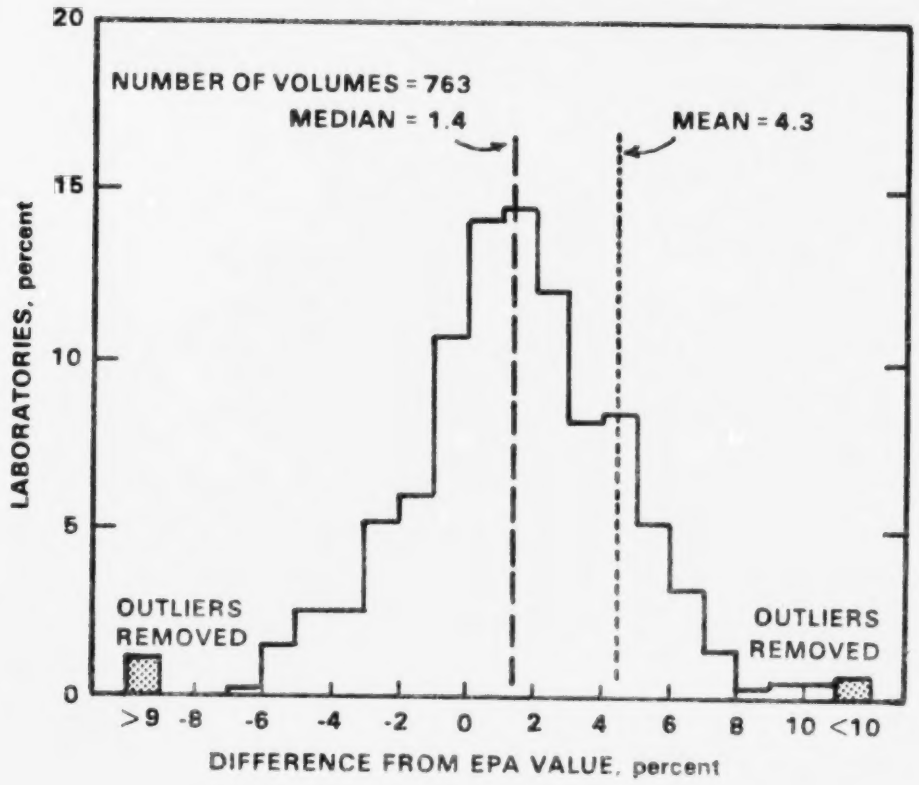


Figure 3. Results of Method 5 audit 0383.

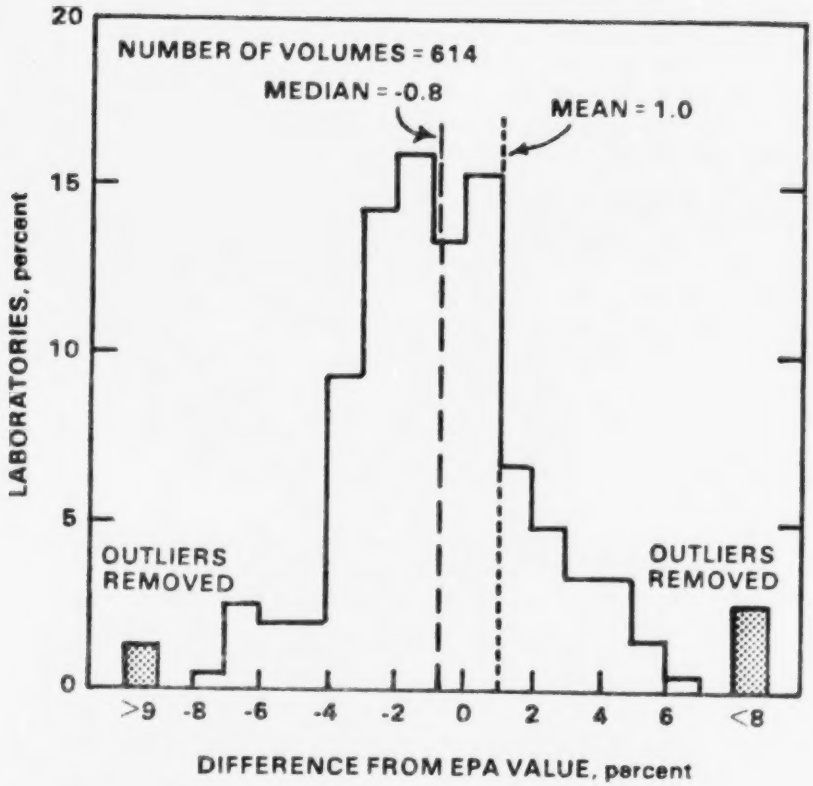


Figure 4. Results of Method 5 audit 0983.

SECTION 4

METHOD 6 AUDIT

This audit checks the participant's ability to analyze Method 6 samples for sulfate. The audit set consists of five dilutions of 10 N sulfuric acid in 25 ml sealed glass ampoules. The analyst withdraws 5.0 ml from each ampoule, adds 30 ml of 3% hydrogen peroxide, and dilutes this sample to 100 ml with distilled water. A 20 ml aliquot is then withdrawn from the diluted sample, 80 ml of 100% isopropanol and thorin indicator are added, and the sample is titrated with barium perchlorate ($Ba[ClO_4]_2$) to a pink end point. In calculating the results, the participants assume an original sample volume of 100 ml, and a sample of 0.021 dry standard cubic meter of stack gas.

Table 7 (which classifies the participants into general categories) compares the total number of participants requesting participation with the number returning data. In the spring audit (0283), 71% of the 160 laboratories that received the audit package returned data. In the fall audit (0883), 61% of the 147 laboratories returned data. Seventy-five laboratories participated in both audits and returned data.

TABLE 7. METHOD 6 AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0283	0883	0283	0883
Contractor	85	76	54	41
Industry	46	44	35	28
Foreign	3	4	2	4
Federal	1	1	1	0
State	16	14	13	11
Local	9	8	8	6
Total	160	147	113	90

Table 8 shows the percentage of laboratories that achieved 2% and 5% accuracy for each of the five different concentrations in the 1983 Method 6 audits. It also shows that 55% of the reporting laboratories in the 0283 audit achieved an accuracy within 2% for the three higher concentrations. However in the 0883 audit, 59% of the laboratories achieved an accuracy

within 2% for all concentrations. Approximately 81% of the laboratories were able to achieve an accuracy level of within 5% on 9 of the 10 samples.

TABLE 8. SUMMARY OF SOURCE SO_2 AUDITS

Concentration	0283		0883	
	± 2%	± 5%	± 2%	± 5%
0 - 500 mg/DSCM	43%	73%	59%	82%
501 - 1000 mg/DSCM	50%	83%	68%	81%
			68%	84%
1001 - 1500 mg/DSCM	58%	84%	61%	84%
			60%	86%
1501 - 2000 mg/DSCM	55%	88%	—	--
2001 - 3000 mg/DSCM	55%	88%	--	--
n	113		90	

SECTION 5

METHOD 7 AUDIT

This audit checks the participant's ability to analyze Method 7 sample for nitrate. The NO_x audit set consists of five dilutions of a potassium nitrate (KNO_3) solution in 25 ml glass ampoules which are autoclaved after sealing so that bacteria which might attack the nitrate are destroyed. The analyst withdraws 5.0 ml from an ampoule, adds this with 25 ml of the Method 7 absorbing solution to a flask, adjusts the pH with sodium hydroxide, and dilutes to 50 ml with distilled water. A 25 ml aliquot is withdrawn from the diluted sample, placed in an evaporating dish, and treated as described in Section 4.3 of Method 7. After this treatment is completed, the absorbance is measured at 410 nm with a calibrated spectrophotometer. In calculating the concentrations present, the participant assumes that 2000 ml of stack gas was sampled.

Table 9 shows both the total number of laboratories requesting participation and the number that returned data for Method 7 audits 0483 and 1083. In the spring audit (0483), 51% of the 124 laboratories receiving the audit package returned data. In the fall audit (1083), 49% of the laboratories returned data. Forty-two laboratories participated in both audits and returned data.

TABLE 9. METHOD 7 AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0483	1083	0483	1083
Contractor	75	67	30	22
Industry	32	31	21	21
Foreign	1	2	1	2
Federal	1	1	1	1
State	8	5	5	4
Local	7	5	5	4
Total	124	111	63	54

The percentage of laboratories that can achieve 5 and 10% accuracy for each of the five concentrations are shown in Table 10. In addition, 32% of the reporting laboratories in the 0483 audit achieved an accuracy within 5% on the lowest concentration. In the 1083 audit, 47% of the laboratories

achieved an accuracy within 5%. Sixty-six percent of the laboratories were able to achieve 10% accuracy on 9 of 10 samples in both audits.

TABLE 10. SUMMARY OF SOURCE NO_x AUDITS

Concentration	0483		1083	
	± 5%	± 10%	± 5%	± 10%
0 - 200 mg/DSCM	32%	51%	47%	66%
201 - 400 mg/DSCM	52%	74%	58%	77%
			63%	83%
401 - 600 mg/DSCM	46%	71%	63%	81%
			42%	73%
601 - 800 mg/DSCM	52%	66%	--	--
801 - 1000 mg/DSCM	52%	68%	--	--
n	63		54	

SECTION 6

METHOD 19A COAL AUDIT

Subpart Da of 40 CFR 60 allows coal sampling and analysis to serve as an acceptable method for demonstrating scrubber inlet flue gas sulfur concentration. The coal audit checks the participant's ability to analyze a coal sample for sulfur, ash, moisture, and BTU content.

Each set of coal samples consisted of two bottles, each containing 50 grams of 60-mesh coal. The participants analyzed each sample for sulfur, moisture, ash, and gross calorific content. The following American Society for Testing and Materials (ASTM) procedures (6) were recommended but not necessarily mandated for participant's use in analyzing the coal samples:

- ASTM D-3177 (Standard Test Method for Total Sulfur in the Analysis of Coal and Coke);
- ASTM D-3174 (Standard Test Method for Ash in the Analysis Sample of Coal and Coke);
- ASTM D-3173 (Test for Moisture in the Analysis Sample of Coal); and
- ASTM D-2015 (Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Method).

The participants measured the parameters and reported their results for moisture (%) on an as-received basis, and their results for sulfur (%), ash (%), and gross calorific value (Btu/lb) on a dry basis.

In the spring audit (0383), 85% of the 130 laboratories that received the audit package returned data. In the fall audit (0983), 84% of the 117 laboratories returned data. Eighty-three laboratories participated in both audits and returned data. Table 11 shows the total number of laboratories requesting participation versus the number that returned data for coal audits 0383 and 0983.

TABLE 11. COAL AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0383	0983	0383	0983
Contractor	72	62	60	51
Industry	41	40	37	34
Federal	1	0	0	0
State	11	11	9	9
Local	5	4	5	4
Total	130	117	111	98

Tables 12 and 13 summarize the coal audit results. The number of analyses is greater than the number of participants because some companies had more than one laboratory participating. In this case, each laboratory received its own set of samples and was asked to analyze the samples in duplicate. Accuracies of 5% and 10% were chosen as the reporting criteria for each of the four parameters (sulfur, moisture, ash, and gross calorific content).

In both audits, 54% of the laboratories were able to analyze the sulfur content of the low level samples within 5% of the expected value. Sixty-three percent achieved 5% accuracy for the high level sulfur concentrations. Only 28% achieved 5% on the low moisture concentration; however, 65% achieved 5% on the higher concentration in the 0383 audit. For the ash analysis and Btu content, 92% to 99% of the reporting laboratories were able to achieve an accuracy within 5% for both sample concentrations.

The participants' accuracy improved with higher concentrations on all parameters except ash, where there was a slight decrease. For those laboratories that reported duplicate analyses, the intra-laboratory precision (repeatability) showed no correlation with concentration level.

TABLE 12. SOURCE COAL AUDIT - 0383

Expected value	No. of analyses	Laboratories accurate within ± 5%	Laboratories accurate within ± 10%
		Sulfur	
0.74%	(1) 125	64%	87%
	(2) 120	64%	83%
2.98%	(1) 127	74%	90%
	(2) 120	71%	86%
		Moisture	
2.05%	(1) 125	34%	69%
	(2) 120	40%	68%
6.13%	(1) 127	65%	87%
	(2) 120	70%	86%
		Ash	
7.40%	(1) 123	96%	99%
	(2) 118	97%	99%
11.42%	(1) 125	92%	100%
	(2) 118	92%	100%
		Gross Calorific	
12719. Btu/lb	(1) 125	94%	99%
	(2) 118	95%	99%
14165. Btu/lb	(1) 123	99%	100%
	(2) 118	99%	100%

TABLE 13. SOURCE COAL AUDIT - 0983

Expected value	No. of analyses	Laboratories accurate within ± 5%	Laboratories accurate within ± 10%
		Sulfur	
0.74%	(1) 112	54%	76%
	(2) 109	56%	77%
2.95%	(1) 113	68%	89%
	(2) 110	63%	88%
		Moisture	
1.62%	(1) 113	29%	48%
	(2) 110	28%	53%
5.75%	(1) 114	26%	74%
	(2) 111	29%	78%
		Ash	
7.31%	(1) 112	97%	99%
	(2) 109	96%	99%
11.33%	(1) 113	96%	99%
	(2) 110	95%	98%
		Gross Calorific	
12470. Btu/lb	(1) 112	97%	100%
	(2) 109	98%	100%
14059. Btu/lb	(1) 111	99%	100%
	(2) 108	99%	100%

SECTION 7

METHOD 3 AUDIT

This audit checks the participant's ability to analyze a gas sample using an Orsat analyzer. The audit package consists of a disposable cylinder that contains a 4 liter sample of oxygen, carbon dioxide, and carbon monoxide. The analyst expels the gas into the Orsat analyzer using positive pressure of the cylinder. The gas sample is analyzed for percentage of carbon dioxide, oxygen, and carbon monoxide.

In the spring audit (0583), 55% of the 110 laboratories receiving the audit package returned data. In the fall audit (1183), 53% of the 94 laboratories returned data. Thirty-seven laboratories participated in both audits and returned data. Table 14 shows the total number of laboratories requesting participation versus the number that returned data for the Method 3 audits 0583 and 1183.

TABLE 14. METHOD 3 AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0583	1183	0583	1183
Contractor	66	52	30	25
Industry	29	24	18	13
Foreign	1	1	1	1
Federal	2	2	2	1
State	9	12	8	7
Local	3	3	2	3
Total	110	94	61	50

Tables 15 and 16 summarize the Method 3 audits. Each laboratory was asked to analyze the sample in duplicate. Five and ten percent accuracy were chosen for the precision reporting criteria for each of the parameters. Each parameter had only one concentration.

In the 0583 audit, only 43% of the reporting laboratories achieved an accuracy within 5% for the CO_2. However, since the CO_2 concentration was higher in the 1183 audit, 61% of the laboratories achieved an accuracy within 5%. In both audits, 81% of the laboratories achieved an accuracy within 5% of the true value for the O_2 analysis which was three times better than

the previous audit (4). For the CO analysis, only 25% of the laboratories in 0583 and 29% in 1183 achieved an accuracy within 5% and 22% of the laboratories did not report a value for CO.

TABLE 15. SOURCE METHOD 3 AUDIT - 0583

Expected value	No. of analyses	Laboratories accurate within ± 5%	Laboratories accurate within ± 10%
		CO_2	
5.00%	(1) 64	44%	73%
	(2) 63	43%	76%
		O_2	
15.10%	(1) 64	84%	91%
	(2) 63	84%	86%
		CO	
1.00%	(1) 53	32%	42%
	(2) 53	21%	43%

TABLE 16. SOURCE METHOD 3 AUDIT - 1183

Expected value	No. of analyses	Laboratories accurate within ± 5	Laboratories accurate within ± 10%
		CO_2	
7.10%	(1) 52	62%	92%
	(2) 51	61%	92%
		O_2	
11.20%	(1) 52	81%	90%
	(2) 51	84%	90%
		CO	
0.50%	(1) 40	35%	35%
	(2) 40	23%	23%

REFERENCES

1. U.S. Environmental Protection Agency. Standards of Performance for New Stationary Sources - Appendix A. Title 40, Part 60, Code of Federal Regulations.

2. Fuerst, R.G., E.W. Streib, and M.R. Midgett. A Summary of the EPA National Source Performance Audit Program - 1980. EPA-600/4-81-077, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, December 1981.

3. Streib, E.W., R.G. Fuerst, and M.R. Midgett. A Summary of the 1981 EPA National Performance Audit Program on Source Measurements. EPA-600/4-83-026, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, June 1983.

4. Streib, E.W. and M.R. Midgett. A Summary of the 1982 EPA National Performance Audit Program on Source Measurements. EPA-600/4-83-049, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, April 1984.

5. Chauvenet, W. Manual of Spherical and Practical Astronomy: Volume II - Theory and Use of Astronomical Instruments (Method of Least Squares). J.P. Lippincott and Co., Philadelphia, Pennsylvania, 1863 (?).

6. American Society for Testing and Materials. Annual Book of ASTM Standards - 1979. Part 26. 01-026079-13, American Society for Testing and Materials, Philadelphia, Pennsylvania.

APPENDIX A

FREQUENCY DISTRIBUTIONS

DGM FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCE - 0383

# Samp.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. dev.	Skewness	Median
763	.0	.4	.8	1.2	1.6	2.2	2.9	3.7	4.4	5.9	466.7	5.7	32.7	12.39	2.2
757	.0	.4	.8	1.2	1.6	2.2	2.9	3.7	4.4	5.8	60.9	3.0	4.3	.00	2.2
754	.0	.4	.8	1.2	1.6	2.2	2.8	3.6	4.4	5.7	14.7	2.8	2.3	-.01	2.2
743	.0	.4	.8	1.2	1.5	2.2	2.7	3.6	4.2	5.6	10.1	2.6	2.0	-.02	2.2
739	.0	.4	.8	1.2	1.5	2.2	2.7	3.6	4.2	5.6	9.3	2.6	2.0	-.04	2.2
737	.0	.4	.8	1.2	1.5	2.2	2.7	3.5	4.2	5.6	8.2	2.6	1.9	-.05	2.2

DGM FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCE - 0983

# Samp.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. dev.	Skewness	Median
614	.0	.3	.7	1.1	1.5	1.9	2.4	2.9	3.5	5.1	303.4	4.1	21.3	13.57	1.9
611	.0	.3	.7	1.1	1.5	1.9	2.3	2.9	3.4	5.0	69.2	2.6	3.7	.02	1.9
609	.0	.3	.7	1.0	1.5	1.9	2.3	2.9	3.4	5.0	14.4	2.4	2.3	-.02	1.9
597	.0	.3	.7	1.0	1.5	1.9	2.3	2.8	3.3	4.8	10.0	2.2	1.8	-.03	1.9
590	.0	.3	.7	1.0	1.5	1.9	2.2	2.8	3.3	4.7	8.0	2.2	1.6	-.05	1.9
589	.0	.3	.7	1.0	1.5	1.9	2.2	2.8	3.3	4.7	7.2	2.1	1.6	-.08	1.9

SO_2 FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE - NO OUTLIERS REMOVED

AUDIT 0283

Sample No.	No. Obs.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
1	113	.06	.35	.82	1.34	1.75	2.39	3.21	4.28	5.89	9.97	296.27	8.76	31.02
2	113	.00	.24	.50	.84	1.39	1.99	2.53	3.49	4.35	8.03	292.08	7.45	29.94
3	113	.01	.30	.36	.69	1.21	1.56	2.08	3.14	3.86	6.65	296.01	6.89	30.02
4	112	.03	.19	.52	.80	1.20	1.91	2.19	2.73	3.83	5.79	294.96	6.62	29.93
5	113	.00	.27	.55	.98	1.40	1.69	2.46	3.38	4.07	6.48	303.47	7.23	30.85

SO_2 FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE - NO OUTLIERS REMOVED

AUDIT 0883

Sample No.	No. Obs.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
1	90	.10	.35	.71	.86	1.03	1.66	2.17	3.43	4.94	12.03	97.00	4.96	11.86
3	90	.00	.11	.43	.59	1.13	1.49	1.98	2.53	3.70	8.38	96.98	4.74	12.95
4	90	.00	.16	.52	.79	1.14	1.33	1.60	2.13	4.52	8.25	96.97	4.91	13.30
6	90	.01	.33	.54	.81	1.27	1.47	1.91	2.91	3.66	5.94	96.97	4.29	12.03
8	90	.02	.20	.39	.72	1.04	1.28	1.48	2.03	3.51	8.71	96.97	3.77	10.69

NO_x FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE - NO OUTLIERS REMOVED

AUDIT 0483

Sample No.	No. Obs.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
1	59	.17	.84	2.51	4.02	6.20	9.38	11.39	14.07	22.95	66.16	772.70	36.91	110.70
2	62	.33	.77	1.37	2.01	3.45	4.45	5.79	8.54	13.46	39.57	638.70	28.64	90.85
3	63	.00	.40	.59	1.63	3.39	5.69	7.28	8.83	15.66	31.41	887.23	32.70	118.83
4	62	.12	.42	1.23	2.01	3.40	4.45	7.84	11.10	16.18	66.48	669.47	30.91	95.03
5	63	.04	.77	1.99	2.34	3.08	4.53	5.83	10.88	15.71	67.13	663.16	32.64	102.22

NO_x FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE - NO OUTLIERS REMOVED

AUDIT 1083

Sample No.	No. Obs.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
2	53	.34	.59	1.60	2.96	3.72	5.15	7.09	11.49	15.63	39.78	98.14	15.32	24.39
3	53	.09	.23	1.56	2.29	3.39	4.35	4.90	8.01	11.72	53.16	111.03	14.92	26.90
5	54	.02	.20	.93	1.57	2.21	2.85	4.10	5.94	8.40	21.76	155.40	13.95	31.62
7	54	.17	.71	1.08	2.12	3.20	4.04	4.74	5.29	8.61	16.77	120.89	13.69	28.76
9	52	.11	.75	1.36	2.51	3.19	5.80	7.08	8.71	11.65	21.94	142.66	15.27	30.32

NATIONAL COAL AUDIT FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES

STUDY: 0383

Sample	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
							SULFUR							
1000	245	.00	.00	1.35	1.35	2.70	2.70	4.05	5.41	8.11	10.81	24.32	5.01	5.30
6000	247	.00	.67	1.01	1.68	2.35	3.02	3.36	4.36	6.04	10.74	35.23	4.37	4.82
							MOISTURE							
1000	245	.00	1.46	2.44	3.90	5.37	6.83	8.29	10.24	14.63	24.39	347.80	12.76	32.22
6000	247	.00	.82	1.47	2.28	2.94	3.59	4.40	5.22	6.85	12.40	38.01	5.71	6.86
							ASH							
1000	241	.00	.14	.27	.54	.81	1.08	1.35	1.62	2.03	3.11	57.97	1.88	5.32
6000	243	.00	.09	.26	.44	.61	.70	.96	1.23	1.49	2.71	8.49	1.31	1.72
							GROSS CALORIFIC							
1000	241	.01	.04	.09	.13	.17	.24	.31	.48	.73	1.57	7.36	.59	.99
6000	243	.00	.08	.13	.19	.28	.37	.50	.72	1.03	2.46	12.66	1.01	1.84

NATIONAL COAL AUDIT FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES

STUDY: 0983

Sample	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
							SULFUR							
2000	223	.00	.68	1.36	1.69	2.71	3.05	4.07	5.42	6.44	10.51	44.41	4.90	6.00
9000	221	.00	.00	1.35	2.70	2.70	4.05	5.41	8.11	13.51	16.22	277.03	9.31	26.92
							MOISTURE							
2000	225	.17	1.57	3.30	5.04	6.61	7.30	8.52	9.57	10.78	12.70	87.83	8.40	9.07
9000	223	.00	1.85	3.70	5.56	7.41	9.88	12.96	16.05	21.60	29.63	91.98	15.12	16.83
							ASH							
2000	223	.00	.26	.44	.62	.88	1.06	1.32	1.59	1.94	2.82	242.54	3.54	22.11
9000	221	.00	.27	.41	.55	.68	.82	1.09	1.23	1.78	2.60	484.40	5.49	45.38
							GROSS CALORIFIC							
2000	221	.01	.30	.59	.80	.96	1.09	1.24	1.49	1.87	2.45	9.59	1.39	1.26
9000	219	.01	.14	.22	.28	.36	.43	.52	.62	.75	1.10	9.17	.63	.87

NATIONAL ORSAT AUDIT FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES

STUDY: 0583

Sample	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
							CO_2							
2000	127	.00	.00	2.00	4.00	4.00	6.00	8.00	8.00	14.00	24.00	98.00	11.15	16.00
							O_2							
2000	127	.00	.66	.66	.66	.66	1.99	1.99	2.65	3.31	10.60	35.10	3.98	6.98
							CO							
2000	106	.00	.00	.00	10.00	10.00	20.00	20.00	40.00	50.00	80.00	80.00	26.13	26.57

NATIONAL ORSAT AUDIT FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES

STUDY: 1183

Sample	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std. Dev.
							CO_2							
3000	103	1.41	1.41	1.41	2.82	4.23	4.23	4.23	7.04	7.04	9.86	43.66	5.87	5.88
							O_2							
3000	103	.00	.00	.00	.89	1.79	1.79	2.63	3.57	4.46	8.93	73.21	4.43	10.01
							CO							
3000	80	.00	.00	.00	20.00	20.00	20.00	20.00	40.00	60.00	60.00	300.00	32.25	43.57

APPENDIX B

INSTRUCTIONS FOR EPA AUDIT MATERIALS

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY
METHOD 5 DRY GAS METER PERFORMANCE TEST DEVICE

NOTE: All procedures referred to are from revised Method 5 published in the Federal Register, Volume 12, Number 160, Part II, Thursday, August 18, 1977, pp. 41776-41782 and references contained therein. This revised method should be adhered to in all details in the use of this quality assurance performance device.

EQUIPMENT: The participant in this study should possess the following equipment, including the performance test device supplied by EPA.

Quantity	Item
1	Method 5/Source Sampling Meter Box
1	Stopwatch, preferably calibrated in decimal minutes
1	Thermometer, ambient range
1	Barometer. If unavailable, call nearest National Weather Service and request the ABSOLUTE barometric pressure. (Corrected for temperature and acceleration due to gravity, but not corrected for altitude.)
1	Performance Test Device. A calibrated flow orifice housed in a quick-connect coupling and identified with an engraved three-digit serial number. WARNING: THE DEVICE MUST NOT BE DISASSEMBLED UNDER ANY CIRCUMSTANCES. Use these devices at room temperature.

PROCEDURE:

1 Calibration of Vacuum Gauge -- The vacuum pressure gauge on the meter box must be calibrated in the range of use (11-22" Hg) against a standard (Hg Manometer) to ensure accurate results.

2 Remove the performance test device from its case and insert it into the gas inlet quick-connect coupling on the source sampling meter box.

3 Turn the power to the meter box on and start the pump.

4 Adjust the coarse flow rate control valve and the fine flow rate control valve to give a reading of 19" Hg (vacuum reading).
CAUTION: The vacuum reading must be accurate and stable for the test period.

5 Allow the orifice and source sampling meter box to warm up for 45 minutes with flow controls adjusted as described in Step 3 before starting quality assurance runs.

PROCEDURE: (continued)

6 Make triplicate quality assurance runs. For each run, record initial and final dry gas meter volumes, dry gas meter inlet and outlet temperatures, internal orifice pressure drop (ΔH), ambient temperature, and barometric pressure. Run duration should be slightly greater than 15 minutes. The following procedure is recommended. Fifteen minutes after a run is started, the participant watches the dry gas meter needle closely. As the needle reaches the zero (12 o'clock) position, the pump and stopwatch are stopped simultaneously. The dry gas meter volume and time are recorded.

This complete run procedure is performed three times to provide the required triplicate quality assurance runs.

7 Calculate the corrected dry gas volume for each run using equation 5.1 of the above-referenced Method 5. For each replicate, record the corrected dry gas volume in dry standard cubic meters, the sampling time in decimal minutes, the barometric pressure in mm Hg, and the ambient temperature in degrees Celcius on the enclosed data card. Be sure to record the performance test device serial number on the data card in the column headed "Orifice Number."

NOTE 1: If you calculate dry gas volume in English Units, use the conversion factor of 0.02832 m^3ft^3 to obtain the volume in metric units.

NOTE 2: If your stopwatch is not in decimal minutes, be sure to convert (e.g. 15 minutes 20 seconds is reported as 15.33 minutes).

8 After recording the requested data on the enclosed data form, return the data form and the performance test device to:

Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711
ATTN: Ellen W. Streib

A postpaid return envelope and label are enclosed for this purpose.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE SO_2 REFERENCE SAMPLES

NOTE: All Method 6 procedures referred to are from the amended method published in the Federal Register Vol. 42, No. 160, Part II, Thursday, August 18, 1977, pp 41782-41784. This amended method should be adhered to in all details in the analysis of these reference standards.

1. Prepare 3 percent hydrogen peroxide according to Section 3.1.3 of the method (30 ml is required for each sample and each blank).

2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampoule and with the ampoule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampoule pipette exactly 5 ml of the reference sample into a 100 ml volumetric flask. Add 30 ml of 3 percent hydrogen peroxide solution. Dilute exactly to the mark with deionized, distilled water. Analyze the sample in accordance with the procedure detailed in Section 4.3 of the method, beginning with "Pipette a 20 ml aliquot of this solution..." (Note: If more than 50 ml of barium perchlorate titrant is required for any sample analysis, a smaller aliquot should be selected to allow titration with less than 50 ml titrant.)

3. Calculate the concentration, C_{SO2} (concentration of sulfur dioxide, dry basis, corrected to standard conditions, mg/dscm), using Equation 6-2. A value of 21×10^{-3} dscm should be used for $V_m(std)$, in the equation. A value of 100 ml should be used for V_{soln} in the equation.

4. Record the reference standard sample numbers and their corresponding SO_2 concentrations in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711
ATTN: Ellen W. Streib

If other than EPA Method 6 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE NO_x REFERENCE SAMPLES

Note: All Method 7 procedures referred to are from the amended method published in the Federal Register Vol. 42, No. 160, Part 11, Thursday, August 18, 1977, pp 41784-41786. This amended method should be adhered to in all details in the analysis of these reference standards.

1. Prepare absorbing solution according to Section 3.1 of the method.

2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampule and with the ampule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampule pipette exactly 5 ml of the reference sample into a 100-ml beaker. Add 25 ml absorbing solution to the beaker; adjust the pH to 9-12 (using pH paper as indicated in Section 4.2 of the method) by dropwise addition of sodium hydroxide (1N). Quantitatively transfer the contents of the beaker to a 50-ml volumetric flask and dilute exactly to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of the diluted sample into a porcelain evaporating dish. Beginning with the evaporation step in Section 4.3, complete the sample analysis.

3. Calculate total µg NO_2 per sample using Equation 7-3. Calculate the sample concentration, C (concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm), using Equation 7-4. A value of 2000 ml should be used for V_{sc} in Equation 7-4.

4. Record the reference sample numbers and their corresponding concentrations, C, in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
Environmental Protection Agency
Research Triangle Park, NC 27711
ATTN: Ellen W. Streib

If other than EPA Method 7 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

COAL AUDIT PROGRAM INFORMATION

1. There is approximately 50 grams of 60 mesh coal per bottle.

2. Analyze the coal samples for moisture and on a dry basis for ash, sulfur and gross calorific value. Report moisture, ash, and sulfur in weight percent with gross calorific value reported as BTU/lb.

3. All methods used in the analysis of these coal samples should follow American Society for Testing and Materials (ASTM) recommended procedures or an accepted automatic analytical device.

4. Suggested procedures are:

Moisture	D-3173
Ash	D-3174
Sulfur	D-3177
Gross Calorific Value ...	D-2015

Please note on the data card (columns 17-32) the ASTM method number. If an ASTM method was not used for analysis note that on the back of the data card. Be parameter specific.

5. If you cannot analyze the coal sample for all four parameters, analyze for what you can. Analysis of moisture is necessary to calculate on a dry basis any of the other three parameters. Analysis of sulfur is also necessary for the calculation of gross calorific value.

6. Analyze each sample in duplicate (if possible) and record results as analysis 1 and analysis 2 for each parameter.

7. Most laboratories will use site number 001. Multiple site numbers are used by laboratories that receive more than one set of samples. These central laboratories have requested auditing of their satellite laboratories.

8. After recording the requested data on the enclosed data card, return the data card to:

Ms. Ellen W. Streib
Quality Assurance Division (MD-77)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

A postpaid return envelope is enclosed for this purpose.

9. If you have any questions concerning this or any source method audit, please call (919/541-7834).

INSTRUCTIONS FOR USING EPA METHOD 3 AUDIT MATERIALS

Equipment Supplied with Audit Kit

(1) Small gas cylinder containing four liters of gas

(2) Nozzle for cylinder (taped on cylinder)

(3) Tygon tubing

Equipment to be Supplied by Participant

(1) Orsat analyzer

(2) Clamp

(3) Extra Tygon or surgical tubing

Procedure

(1) Leak-check apparatus by clamping off tubing. SEE DIAGRAM.

(2) Fill up burette with gas by using positive pressure. Vent this sample through the manifold to the atmosphere.

(3) Repeat Step 2.

(4) Fill up burette past fill mark with the gas and carefully vent out excess to the atmosphere, until the fill mark is reached.

(5) Analyze for CO_2, O_2 and CO as described in Sections 4.2.5, 4.2.6 and 4.2.7 of EPA Method 3.

(6) Record the results on the data card enclosed with the sample.

(7) Repeat Steps 4 through 6.

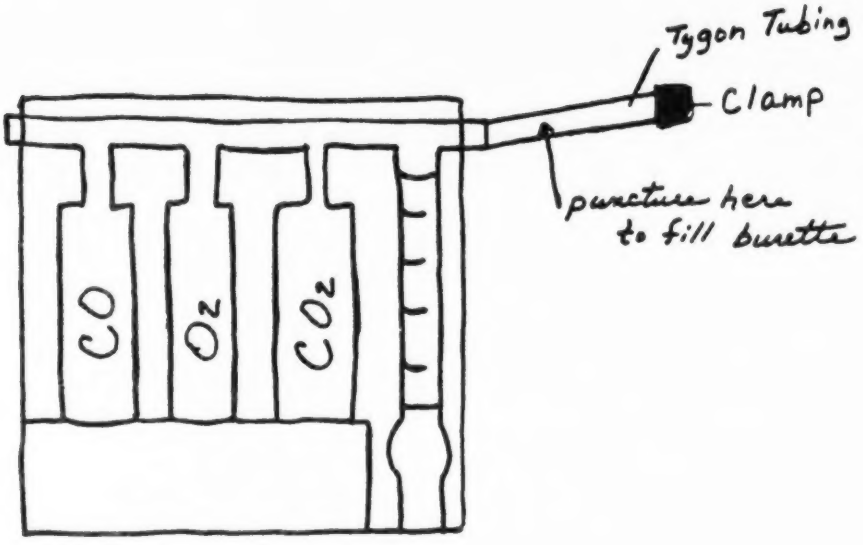
CAUTION: If the tubing is punctured excess times, leakage can occur. Replace if necessary.

Send the data card to the address below. (The cylinder gas can should not be returned.)

Ms. Ellen Streib
Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

Note: Site number will always be 001 except when other Orsat apparatus or participants are using the same gas sample. The extra apparatus or participants should be labeled 002, 003, etc.

APPARATUS SET-UP



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